ASSESSMENT OF SPONTANEOUS HEATING LIABILITY OF SOME INDIAN COAL BY CROSSING POINT TEMPERATURE AND OLPINSKI INDEX METHOD

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY
IN
MINING ENGINEERING

BY

DEBANANDA NAYAK
109MN0012

DEPARTMENT OF MINING ENGINEERING

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CERTIFICATE

This is to certify that the thesis entitled **"ASSESSMENT OF SPONTANEOUS HEATING LIABILITY OF SOME INDIAN COAL BY CROSSING POINT TEMPERATURE AND OLPINSKI INDEX METHOD"** submitted by **Debananda Nayak (Roll no. 109MN0012)** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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Debananda Nayak

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ABSTRACT

Carbonaceous Organic Aromatic Lump (COAL) is a heterogeneous and stratified Organic rock, formed from decay of vegetable matters or sub-merging forest due to tectonic movement with carbon content varies from 60 to 100%. According to carbon content, Coals are classified in different stages like Peat (50.9%), Lignite (61.8%), Sub-Bituminous (64.5%), Semi-Bituminous (69.5%), Bituminous (78.5%), Anthracite (91%), keeping in view that Graphite (100%) will be the last stage of formation. As the major elemental constituents of coal are Carbon, Hydrogen, Nitrogen, and Oxygen and Sulphur so when these coals are exposed to open-air, it absorbs Oxygen from atmospheric air, liberating heat and ultimately leading to fire. As this liberation of heat is due to the characteristics of Coal, so it is known Spontaneous Heating or Auto-oxidation of Coal or Endogenous Fires which extends during mining. The propensity of this fire is so large noxious gases like CO, CO_2, SO_2, Nitrous fumes and ultimately, mining activity stops, by blocking up the huge amount of capital and sterilization of reserve [18].

This project deals with the assessment of propensity of different coal samples towards spontaneous heating by assessment of various constraints determined by experimental procedures. The intrinsic properties as well as susceptibility indices of the coal samples have factually been reflected to predict self-heating liability of coal. Four coal samples were collected from MCL (Mahanadi Coalfields Limited) and five from SECL (South Eastern Coalfields Limited), covering some coal fields of India. Liability of collected coal samples were determined using Crossing point temperature and Olpinski index apparatus and the intrinsic properties were determined using proximate analysis method following Indian standards. Based on the results correlation analysis was done among the parameters of proximate analysis and CPT and Sz_a, also found out the best correlation among them.

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CHAPTER-1

INTRODUCTION

1.1 BACKGROUND

Coal is the prevailing energy supply source in India, accounting for about 67% of the total energy consumption in the country. About 70% of coal production is used for power generation in India .Therefore care should be taken to prevent the loss of coal by various causes. Around 34% of electricity produced in the world. Indian coal found to contain high ash content and low calorific value. Mine fires in Indian coalfields is generally induced by spontaneous heating of coal [20].

Fig. 1.1 Coal mines in India [6]

Now a day's various preventive technologies being adopted in order to determine the liability of coal to spontaneous heating different methods have been followed by various researchers of the world. A number of experiments have been performed and formulated for assessments of spontaneous heating susceptibility of coal viz., Differential thermal analysis, Crossing point temperature method, Olpinski index method , Wet oxidation potential technique, Flammability temperature. A number of approaches have been developed over the years to assess the susceptibility of coal towards spontaneous heating. This liability to self-heating of coal also resolves the incubation period of coal seam, which decide the dimension of the panel to be created, which is a most vital safety should be taken in account while mine scheduling. It is therefore imperious that the persons making plan for a mine define in advance the spontaneous heating susceptibility of the seam to be mined so that either the entire coal has been taken out before the incubation period of the coal, or advance precautions are planned to tackle this problem and prevent spontaneous heating [20].

1.2 OBJECTIVE OF THE PROJECT

The objective of this project is to carry out comparison among all the coal samples collected from MCL and SECL based on the susceptibility of coal to spontaneous heating. The project is subcategorized into various segments, which are shown below.

1.2.1 Literature review

Collection of all the past works of various researchers both national and international as well as gathering information about the topic from journals, proceedings, books etc.

1.2.2 Sample collection and preparation

Four samples were collected from MCL and five from SECL and prepared as per requirement of the individual experiments following Indian standards.

1.2.3 Experimentation

The experimental investigations are divided into two stages:

1. Determination of intrinsic properties of coal – proximate analysis.

2. Determination of susceptibility indices of coal – Crossing point temperature and standard Olpinski index.

1.2.4 Analysis

Correlation analysis was done using MS Excel package among the various parameters of proximate analysis and the susceptibility indices obtained to find out the best correlation among them. The effect of each parameter and indices are evaluated.

CHAPTER – 2

LITERATURE REVIEW

2.1 COAL MINE FIRES

It is related with typically coal mines, through fires in pyrite mines and is general happenings in coal mines but is unusual in metal mines. A study of the reasons of coal mine fires reflects that they may start either from an open fire initiate because of the heavy nature of coal. Mine fires can be prompted either by spontaneous heating, electrical failures, explosion of gases and blasting in mining operation. In coal mines the major reason of mine fire is spontaneous heating of coal. The process in which the coal catches fire automatically on coming in impinging with oxygen in the atmosphere without any outside source of fire which leads to mine fires is acknowledged as spontaneous heating of coal [17].

2.1.1 ENDOGENOUS FIRES [17]

1. **Pyrite fires:** As coal pyrite also reacts with oxygen present in air at room temperature releasing heat which under advantageous conditions of heat amassing gives rise to spontaneous fires. Proneness to spontaneous heating of pyrites is much less than that of coal but it increases if carbonaceous materials are present in pyrites. There are occurrences when pyrites with 5 - 6 % C and 10 – 12 % S have caused fire due to spontaneous combustion.

2**. Endogenous heating of timber:** Decayed timber or rotten wood may under very advantageous circumstances give rise to spontaneous heating assumed to be mainly from bacterial origin.

2.1.2 EXOGENOUS FIRE [17]

Electricity is one of the main reasons of mine fires. It may initiate from short circuit of electric wires used, heat generated out of machineries, flames from ignition, candles or explosion while blasting and ignition of inflammable gases and materials like timber, oil or wastes. At the time of processing of sulphide ores or fires from surface.

2.2 HISTORY OF COAL MINE FIRES

Self-heating of coal spreads to spontaneous heating is the most considerable reason of fires in coal mines through the world. Spontaneous heating of coal can occur in underground coal mines, coal stockpiles, opencast mines, transportation and during the disposal of barrens from coal applying industries in heap wastes [12].

World scenario [13]

Up to 10 coal fires per year in the Ruhr area of Germany are triggered by self-heating of coal. In china underground fire due to spontaneous heating of coal is extended within a region stretching 5000 Km about east – west and 750 Km about north – south. It is assumed that fires in northern China consume an estimated amount of 100–200 MT of underground coal which is about 2–3 % of world CO_2 production .surveys in the West Riding of Yorkshire (England) showed that 45 of the county's 153 collieries were on fire in 1931. A more recent example is the spontaneous combustion of spoil heaps at Middleburg colliery in Witbank coalfield in South Africa.

China

For China, which is the world‘s biggest coal producer having an yearly production around 2.5 billion tons, coal fires are a dangerous problem. It has been evaluated that some 10-20 million tons of coal uselessly burned away annually, and that the same quantity again is built unreachable to mining. They are centered in provinces of Xinjiang and Inner Mongolia and Ningxia. except exits from burned and inaccessible coal, these fires add to pollution of air and significantly increased levels greenhouse gas ejecting and have thereby develop a problem which has grew international attention.

United States

Most of coalfields in the USA are affected by spontaneous ignition. The Federal Office of Surface Mining (OSM) keeps a database (AMLIS), which in 1999 recorded 150 fire zones. Pennsylvania having 45 fire zones, the most well-known being the fire in the Centralia mine in the hard coal area of Columbia County. In Colorado coal fires have raised as a result of variations in the groundwater level, which can raise the temperature of the coal up to 30°C, enough to initiate spontaneous heating. The Powder River Basin in Wyoming and Montana comprises of some 800 billion tons coal, and already the Lewis and Clark Expedition stated fires there.

Germany

In Planitz, a part of the city of Zwickau, a coal seam caught fire and had been burning since 1476 could only be controlled in 1860. In Dudweiler (Saarland) a coal seam fire exploded around 1668 and is still burning till date. Also well-known Stinksteinwand (stinking stone wall) in Schwalbenthal on the eastern slope of the Hoher Meibner, where different seams caught fire long ago after lignite coal mining stopped; gas produced during combustion continues to reach the surface in these days.

Indian scenario [13]

History of coal mine fires can be drew back to the year 1865, when the first fire was defined in Raniganj Coalfields. Over 140 years fire have been stated till the year1967 from both Jharia and Raniganj coal fields and higher-up class of non-cocking coal in Raniganj coal fields. Fire appears whenever and wherever burnable material is present in mine working. They threaten not only worthful lives of men in mines but also induce considerable economic losses to the association. These fires are not only affecting border areas, adding to the losses but also combat economic mistreatment of the seam in the locality. Again open fires in these fields induce environmental pollution by emission of vast quantities of steam, smoke and deadly gases causing a serious health hazards. The main features of starting the fire in India are that the coal seams are thicker and there is a propensity of spontaneous heating during the extraction operation. The problem of extraction of thick seam and coal standing in pillars is a dangerous one particularly in cases where they are with prominent moisture, high volatile and low ash which is more liable to spontaneous combustion of coal. It is not possible to extract all the coal by caving method or even by complete packing under Indian mining circumstance. Pillars standing for long time are responsible to devolve in straight and slopping may occur.

2.3 MECHANISM OF SPONTANOEUS HEATING

The auto oxidation of coal is a complex physico-chemical process which is consists of a series of event like oxygen absorption, coal-oxygen complexes formation and their decomposition leading to the discharge of heat. This complexity of the process is vast because of the great diversity in the coal substance with the associated mineral matter and the conditions of oxidation. During the oxidation of the heterogeneous mass, concurrent and overlapping reactions take place which are very difficult to separate out. The rate of oxidation at ambient temperature gives a measure of the proneness of coal to auto-oxidation. This low temperature aerial oxidation of coal is not a singular chemical process but a complex phenomenon generally comprising of several simultaneous and interesting chemical processes which result both in erosive material removal and structural alteration of the organic matter. A large number of stable chemical chain reactions take place due to several oxidation states of carbon and a variety of strong carbon oxygen complex is formed. The noticeable compositional, elemental and structural variations reveal that the reaction of oxygen with solid coal is a time dependent dynamic method. Noticeable variations in coal molecular structure and composition arise from prolonged sequence of events whose components exhibits complex inter-relationships. Thus the reaction environment is heterogeneous intrinsically because two bulk phases, solid and gas are present and extrinsically because various structural changes brought by the reaction, affect the overall coal reactivity. Porous solid absorbs the liquids or gases or the solution of gas/liquid, which is known as sorption. When accumulation restricts at the surface liberating heat and rate of penetration is negligible then it is considered as adsorption but if uniform penetration in the bulk of the solid occurs, then it is called absorption. The absorptions is always an endothermic phenomenon and starts from the surface of the solid and consumes heat of the solid for penetration. The energy on the surface is always low. The method where physical forces like inter-molecular attraction are blamable, are known as physical adsorption or Van der Wall's adsorption, but when operative force needs to break the chemical nature of the compound is called chemisorption or activated adsorption. This physical adsorption is predominant at very low temp whereas chemical adsorption is very low at low temp whereas rate of reaction increases with increase in temperature. Coal gets heated up on absorbing oxygen, whose decomposition phenomenon can be expressed/explained in the following manner. Oxidation is very slow below 50^oC and accelerates above 50^oC, but above 80^oC, a period of steady state is maintained, probably due to the removal of moisture of coal. The removal of oxides of carbon occurs from 120^oC. The interaction of oxygen with coal accelerates rapidly up to 180^oC and thermal decomposition starts between 180^oC to 220^oC. Self-sustained process of combustion begins in between 220^oC to 275^oC with very rapid rise of temperature until the ignition point is attained [2].

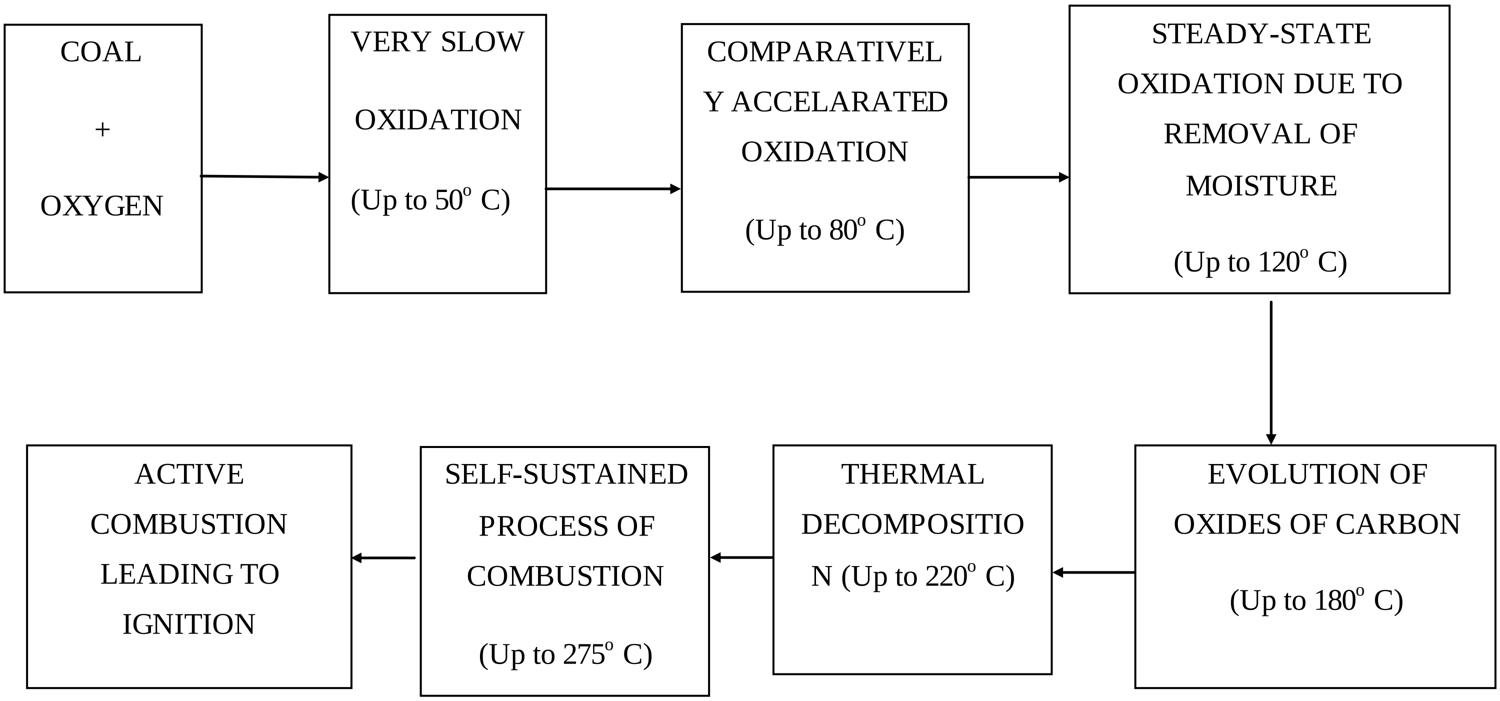

Fig. 2.1 Sequential stages in the spontaneous combustion of coal [2]

2.4 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL [4]

The main cause for the problems in accepting the mechanism of spontaneous combustion is the presence of many internal and external factors affecting the initiation and development of the phenomenon. These factors have been reviewed by numerous researchers. The main reasons which have substantial effects on the method are shown in the table 2.1.

Table 2.1 Factors affecting spontaneous combustion of coal [4]

Intrinsic Factors (Nature of Coal)	**Extrinsic Factors (Atmospheric, Geological and Mining Conditions)**
Pyrites	Temperature
Moisture	Moisture
Particle size and surface area	Barometric pressure
Rank and petrographic constituents	Oxygen concentration
	Bacteria
Chemical constituents	Coal seam and surrounding strata
Mineral matter	Method of working
Petrographic composition	Ventilation system and air flow rate
Volatile matter	Timbering
Rank of coal	Roadways

2.5 THEORIES OF SPONTANEOUS COMBUSTION OF COAL

Pyrite theory

Combustion because of oxidation of pyrites has been recognized to be a common process in pyrite mines. Heating of coal can be initiated by iron pyrites (only when present in substantial proportion) and in finely powdered and spread state in the existence of moisture. The reaction between iron pyrites, oxygen and moisture is exothermic, hence yields materials of greater volume than the original pyrite thus availing more pore for oxygen to be absorbed. The reaction can be shown as

$$2Fe\ S_2 + 70_2 + 16H_2O = 2\ H_2SO_4 + 2\ Fe\ S0_4.7H_2O + 316\ kcal$$

The oxidation reaction of pyrite during weathering of coal seam may be shown by

$$Fe\ S_2 + 15/4\ O_2 + 9/2\ H_2O = Fe\ O.OH + 2\ H_2SO_4$$

The equation shows that both oxygen and moisture, two main weathering causes, contribute to pyrite alteration and the sulphuric acid is made as a by-product of the change. In comparison to dry coals in the existence of moisture, the reactivity of coal is found to be double and if pyrite is in finely spread state, it becomes 10 fold. It was seen that pyrite below 5% showed worthless effect [2].

Bacterial Theory

Bacteria were also assumed to encourage self-ignition of coal. Later examinations revealed that bacteria had little effect on the self-heating tendency of coal. The involvement of heating due to the action of bacteria cannot be totally ruled out. Spontaneous heating witnessed in haystacks and in wood are known to be mainly due to bacterial action. However, there is no vital proof to authenticate or cast out this theory [2].

Humidity theory

It says the quantity of heat liberated by atmospheric oxidation of coal is much less than the amount of heat required removing water from the coal. If the evaporation of water can be induced at the seat of heating, then the temperature of heating would decline. When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above system well explains other possible sources of CO and CO_2 in low temperature reaction between coal and oxygen. Nordon et al (1979) measured the heat of wetting with water of an Australian low rank bituminous coal with different moisture content. Although dry coal showed a substantial rise of temperature, heat of wetting (6 KJ / Kg) values decreases rapidly with increasing moisture content and heat. For a normal moist coal (65% relative humidity) heat of wetting could cause a temperature rise of only 2°C, which would be unlikely to contribute importantly to self-heating in store coal [9].

Coal-oxygen Complex Theory

Oxidation of coal is believed to be initiated at native radical sight. Formation of peroxy radical and hydro peroxides is commonly to be thought to be they mechanism by which oxygen and moisture are initially in corporate into organic matrix. These species may react, rearrange or decayed to form wide range of oxygen functionality in the matrix or gaseous product [2].

2.6 LITERATURE REVIEW

National status:

Banerjee et al. (1972) determined the Crossing Point Temperature (CPT) of a number of Indian coal samples adopting the Crossing Point Temperature method. He found that coals with crossing point temperatures above 160°C are badly susceptible. Between 120^0C & 140^0C could be considered to be extremely susceptible to spontaneous heating.

Nandy et al. (1972) illustrated the variation in Crossing Point Temperature values with the, oxygen percentage, moisture content and the volatile matter of coal. He detected that CPT normally minimizes with maximizes in each of these constituents of coals. But outside 35% V.M, 9% oxygen, or 4 to 6% moisture content there is not much change in CPT values. In fact, above 4 to 6% moisture content in coal, then CPT values shows a developing trend.

Banerjee (1985) derived the formula for the Olpinski index, in this method small pellet of coal is allowed to undergo aerial oxidation at temperature around 135^oC gives the measure of spontaneous heating susceptibility S_{Za} of the concerned coal. Olpinski method makes correction for ash content of the coal (A), and expressed spontaneous heating index as: $S_{Zb} = (S_{Za} / (100-A))*100$

Karmakar et al. (1989) observed the three methods for determining spontaneous combustion susceptibility of cola have been sorted out the experimental data obtained by these methods for sixteen coal samples. This assessment concludes that a linear relationship exist amongst CPT, polish Sz_a and Russian U indices. Coal with low moisture and high VM content have high susceptibility to spontaneous combustion. S_{Za} index being a convenient and quick method can be applied as a substitute to CPT method now being complied in India. In this method a small pellet of coal is appropriated to undergo aerial oxidation at a temperature about 230°C with the help of quinoline liquid.

Panigrahi et al. (1996) carried out work on wet oxidation and CPT with respect to intrinsic properties of coal samples covering most of the coalfields of India and determine the susceptibility of coal to spontaneous heating.

Nimaje et al. (2010) made thermal studies on spontaneous heating of coal. Of all the experimental techniques developed thermal studies play an important and dominant role in assessing the spontaneous heating susceptibility of coal. They made an overview of thermal studies carried out by different researchers across the globe for determination of spontaneous heating of coal and revealed that lot of emphasis on experimental techniques is necessary for evolving appropriate strategies and effective plans in advance to prevent occurrence and spread of fire.

International status:

Olpinski et al. (1953) observed the exothermicity of the coal pellet at 235^0C provides the spontaneous heating susceptibility of the coal and the electronic recorder recorded time-temperature curve of the coal bed crosses 235^0C. The rate of rise of temperature at 235^0C provides Olpinski index.

Feng et al. (1973) discovered a composite liability index using the results of Crossing Point Temperature experiment s, called FCC index. This is calculated using the following equation

$$Liability\ Index = \frac{Average\ heating\ rate\ between\ 110°C\ and\ 220°C}{Relative\ investigation\ temperature} * 100$$

Kaymakci et al. (2002) used linear and multiple regression analyses to determine the relationship between spontaneous combustion parameters (derived from time temperature curves obtained from laboratory tests) and coal parameters (obtained from proximate, ultimate and petrographic analyses). The linear regression analyses have shown that ash (A), volatile matter (VM), carbon (C), hydrogen (H), exinite (E), inertinite (I) and mineral matter (MM) are the major factors that affect spontaneous combustion.

Singh et al. (2007) observed in opencast mines, coal immediately oxidises and catches fire due to the intrinsic characteristics of coal, such as high moisture, low rank, high volatile matter, and presence of sulfur in the form of pyrites, low crossing point temperature (CPT) and ignition point temperature (IPT) value and less incubation period. In surface mines, when the coal benches are left idle for a longer period, heat addition takes place in favourable conditions and sometimes results into fire. The objective of this paper is to present the different successful case studies regarding the safety management of open pit coal mines from occurrences of spontaneous heating.

CHAPTER – 3

EXPERIMENTAL INVESTIGATION

3.1 SAMPLE COLLECTION AND PREPARATION

3.1.1 SAMPLE COLLECTION

It is the process by which the physical and chemical properties of the mineral or ore can be observed with the preferred accuracy. It is the method of collecting the small portions of a whole such that consistence of that portion presents that of entire mass .Various types of sampling are:

a. Channel sampling

b. Chip sampling

c. Grab sampling

d. Bulk sampling

e. Drill hole sampling

Out of the sampling methods mentioned above, channel sampling method is the common method generally accepted to collect the coal sample from the insitu coal seams following the Indian standard..

Channel sampling

The part of seam to be tested shall be exposed from the roof to the floor. The same sample shall be taken in a channel representing the entire cross-section of the seam having the dimensions of 30x10cm, that is, 30cm in width and 10cm in depth. For this purpose, two parallel lines, 30cm apart at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, newly exposed surface of the seam. Obvious dirt bands exceeding 10cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimized [8].

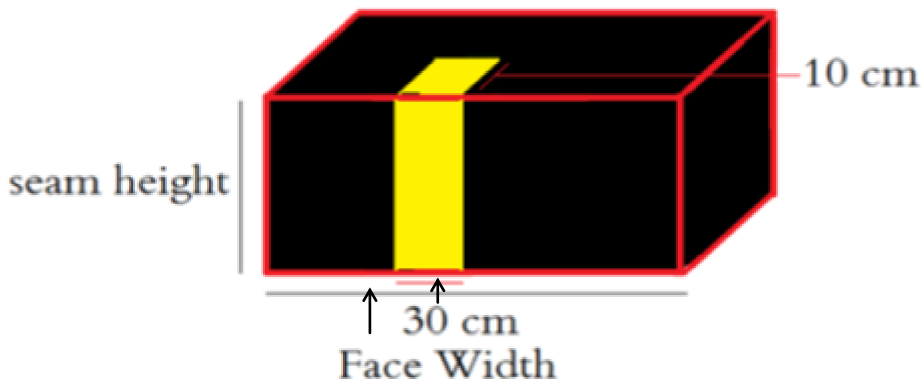

Fig. 3.1 Channel Sampling [15]

3.1.2 SAMPLE PREPARATION

The samples received from the field via channel sampling methods are crushed in the laboratory as per the experimental requirements. The crushed sample is then sieved to required sizes and stored in air tight polythene packets. The packets are stored in air tight containers for further use in experimentation. Samples were prepared according to IS 436 Part 1, Section 1-1964 and IS 436 Part II-1965 [8].

3.2 PROXIMATE ANALYSIS

Proximate analysis was developed as a simple mean of determining the distribution of products found of coal. When the coal sample is heated under specified conditions, then it classifies the products into four groups: i) moisture; ii) volatile matter iii) fixed carbon, iv) ash, the inorganic residue remaining after combustion. For proximate analysis, i.e. for the determination of volatile matter, moisture, ash and fixed carbon, the method determined by IS (Indian standard) 1350 (Part-I) – 1984 was followed [7].

3.2.1 Determination of moisture content (M)

Coal is always associated with some amount of moisture, which is both physically and chemically bound, due to its nature, origin and occurrence. It is customary to differentiate between extraneous and inherent moisture. When a wet coal is exhibited to atmosphere, the external moisture evaporates, but the obviously dry coal still contains some moisture, which can be removed only on heating above 100^0C. External moisture is also called accidental or free moisture, whereas inherent moisture is named as equilibrium or air-dried or hygroscopic moisture. The quantity of external moisture counts mainly on the mode of occurrence and handling of coal, but the air-dried moisture is associated to the inherent hygroscopic nature of the coal.

Procedure

1. About 1g of finely pulverized -212 micron air-dried coal sample is weighed in a silica crucible and then placed within an electric hot air oven and is maintained at 110^0C.
2. The crucible with the coal sample is allowed to put in the oven for 1.5 hours and it is taken out with the help of tongues, and then cooled in a desiccator for about 15 minutes.
3. Then weighed and loss in weight is reported as moisture (on percentage basis).
4. The calculation is done as per the following.

$$\textbf{Moisture \%} = \frac{\mathbf{Y-Z}}{\mathbf{Y-X}} * \mathbf{100}$$

Where,

X - Weight of empty crucible, in grams (g)

Y - Weight of crucible and coal sample before heating, in grams (g)

Z - Weight of crucible and coal sample after heating, in grams (g)

3.2.2 Determination of volatile Matter (VM)

When coal is heated in defined equipment under appointed conditions, is concerned to as volatile matter, the loss of mass and corrected for moisture. The matter lost is composed of materials that form upon the thermal decomposition of the various constituents of coal. Some of the element of coal volatile matter is hydrogen, carbon monoxide, methane and other hydrocarbons, tar vapors, ammonia, some organic sulphur and oxygen containing deepens and some incombustible gases, such as carbon dioxide and water vapour, all of which come from the decomposition of organic materials in coal. Inorganic materials in coal contribute the water of hydration of mineral matter, carbon dioxide from carbonates and hydrogen chloride from inorganic chlorides to the volatile matter.

Procedure

For determining the volatile matter a special volatile matter silica crucible (38 mm height, 25 mm external diameter and 22 mm internal diameter) was used.

1. First the empty silica crucible along with the lid uncovered was heated at 800^0C for an hour in the muffle furnace and then cooled to room temperature.
2. The empty volatile matter crucible was then weighed again.
3. Approximately 1g of coal sample was weighed in the volatile matter crucible and it was placed inside the muffle furnace maintained at 925^0C with the lid covering the crucible.
4. The heating was carried out exactly for 7 minutes, after which the crucible was removed, cooled in air and then in a desiccator and weighed again.

$$\textbf{Volatile matter } \% = \frac{Y - Z}{Y - X} * 100 - M\%$$

Where X = weight of empty crucible, in grams (g)

Y = weight of crucible and coal sample before heating, in grams (g)

Z = weight of crucible and coal sample after heating, in grams (g)

M%=Moisture percentage

3.2.3 Determination of Ash (A)

During the ashing process, the coal ash is the residue left after the combustion of coal under defined conditions. It does not occur as such in the coal, but is formed as the result of chemical changes that take place in the mineral matter. Ash and mineral matter of coal are therefore not identical .The extraneous and inherent mineral matters are the two types of ash forming materials in coal. The extraneous mineral matter consists of materials like calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum.

Procedure

1. Weight of the empty crucible is taken.
2. 1g of desired coal sample is weighed in the crucible and is taken in a muffle furnace at 450^0C
3. For 30 minutes and the temperature of the furnace is raised to 850^0C for 60 minutes.
4. After that time interval, the crucible is taken out and placed in a desiccator and weighed.

$$\textbf{Ash}\% = \frac{Z-X}{Y-X} * 100$$

Where,

X= weight of empty crucible in grams (g)

Y= weight of coal sample and crucible in grams (g) before heating

Z= weight of coal sample and crucible in grams (g) after heating

3.2.4 Determination of Fixed Carbon (FC)

The materials remaining after the determination of moisture, volatile matter and ash is fixed carbon by definition. Fixed carbon plus ash present the approximate yield of coke from coal. The value of fixed carbon is determined by subtracting from 100 the resultant summation of moisture (M), volatile matter (VM) and ash (A).With all portion on the same moisture reference basis.

FC=100-(M+VM+A)

3.3 CROSSING POINT TEMPERATURE METHOD

This is one of the oldest approaches for determining susceptibility towards spontaneous heating of coal. It is the temperature at which the coal temperature coincides with that of the furnace temperature or bath temperature in °C. In this method, the coal sample is heated in a furnace within a reaction tube at constant rising temperature with oxygen passing through it at a predetermined rate till the coal temperature crosses the furnace temperature [17].

Procedure [2]

The setup for the determination of crossing point temperature (CPT) of coal consists of following: Vertical tubular furnace which has an internal diameter of 50 mm and a heating capacity of 3KW. The furnace is provided with a temperature controller. Glass reaction tube is of 26 mm internal diameter and 150 mm in length. The reaction tube has spiraling glass tube of 6 mm internal diameter around it which is connected to the bottom (inside) of the reaction tube for air inlet and a small out-let tube at the top acts as air/gas outlet.

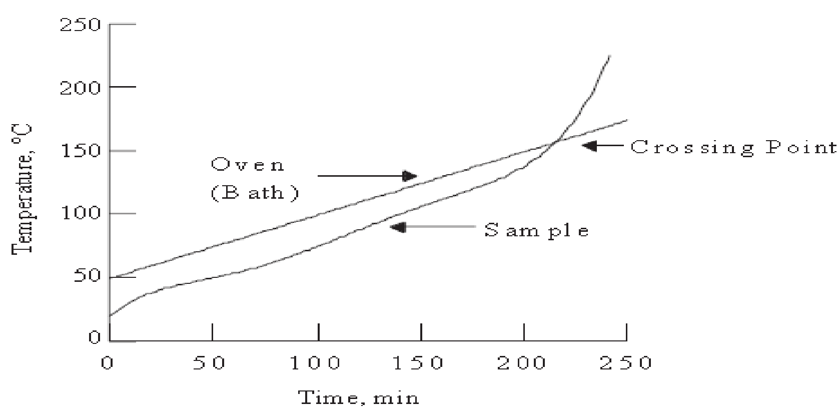

Fig. 3.2 Time Vs. Temperature curve of CPT [4]

A sulphuric acid bubbler is used to remove moisture in air. A drying tower containing granular calcium chlorides is used to remove moisture from air. 4g sample of size -100+200 mesh (Dp = 112 micron) was placed in the reaction tube followed by glass wool at the bottom most position and a small sieve of 200 mesh (Dp = 72 micron) on that. The tube is then lightly tapped a fixed number of times to achieve uniform packing density of the samples. The reaction tube is then placed in the tubular furnace and a chromel-alumel thermocouple is inserted at the center of the sample. The entrapped air and occulted gases are removed from the coal samples by passing a mild current of nitrogen through the sample for three minutes, without disturbing the packing. The furnace is switched on and simultaneously oxygen is allowed to pass through the sample, with an average rate heating of 1^0C per minute and at 80 ml/ min. the temperature of the furnace (bath) and the coal sample are recorded at every five minute interval till the temperature of coal crossed over and gone beyond the furnace temperature [2].

Table 3.1 Classification of CPT [12]

CPT(^{0}C)	Risk Rating
120-140	Highly susceptible
140-160	Moderately susceptible
>160	Poorly susceptible

Plate. 3.1 Crossing point temperature set-up

3.4 **OLPINSKI INDEX METHOD** [10]

In this method, liquid quinoline is heated in an electric oven to boil gently at a temperature of 230^{0}C producing quinoline vapour. The coal samples are powdered and small pellets of 1g of -72 mesh are prepared out of these powdered samples. These pellets are heated indirectly by quinoline vapour in an atmosphere of oxygen which is made to flow over the coal pellet at predetermined rate. While heating the coal pellets, a thermocouple is inserted into it and output of the thermocouple is given to a temperature recording system. The temperature versus time plot of the coal samples is directly obtained by this instrument. The rate of rise of temperature at the moment of equalization of supplied oxygen gas and coal pellet temperature is either graphically determined by drawing tangent to the curve at the point corresponding to the quinoline vapour temperature at 230^{0}C or the first derivative of the curve at 230^{0}C represents the slope of the curve at the same temperature. The rate of rise of temperature determined in this way is expressed in ^{0}C/min is an indication of spontaneous heating susceptibility of the coal .This index is known as Olpinski index and is denoted by Sz_a. In this method Sz_a index is corrected for ash content of the coal and is expressed as Sz_b which is as given below:

$$Sz_b = (Sz_a\ (100\text{-}A))*100$$

Where, Sz_b – Spontaneous heating index free of ash

A – Ash content of coal expressed in %.

The increase of Sz_b index indicates that the sample is more susceptible to spontaneous combustion.

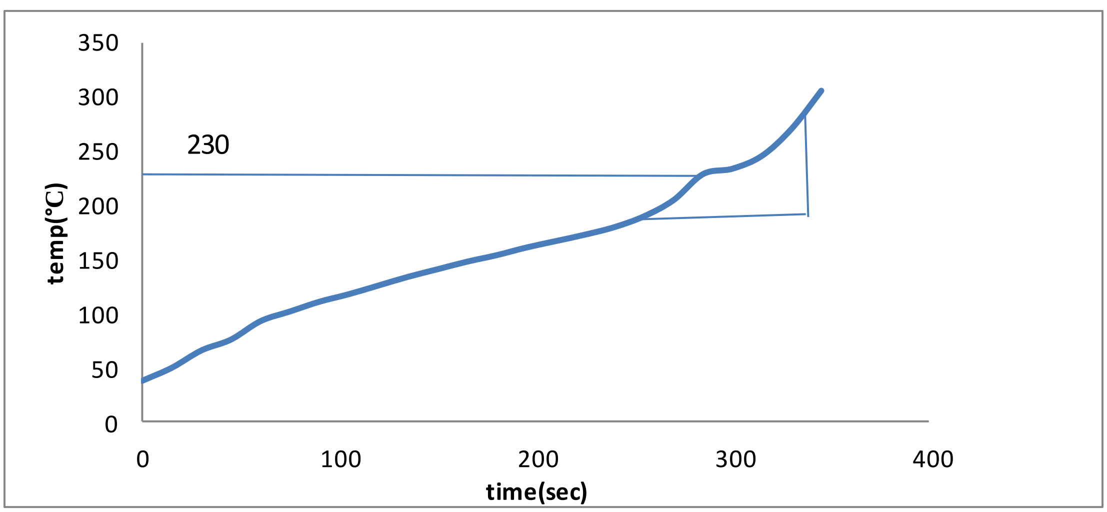

Fig. 3.3 Olpinski curve

Plate. 3.2 Olpinski index apparatus

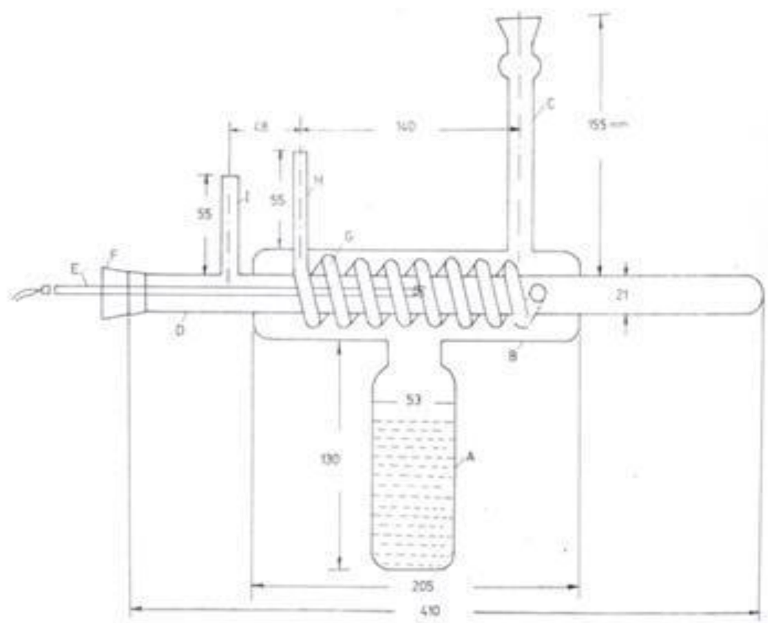

Fig. 3.4 Schematic diagram of Olpinski index apparatus [11]

The apparatus consists of a cylindrical glass vessel (A) of volume about 280 cc which is connected to another cylinder (B) perpendicular to it. One funnel (C) is attached to B which also acts as a condenser. A reaction tube (D) passes axially through B which can be heated by quinoline vapour. One end of a thermocouple (E) can be introduced to reach up to the central line of quinoline vessel. A glass tube (G) goes spirally over the reaction tube to release at its closed end. The combustion product goes out through the outlet (I). The apparatus is mounted over the electric oven to the heat quinoline vessel. The whole set-up placed in a fume chamber with an exhaust fan to clear the quinoline vapour out of the laboratory [10].

Table 3.2 Classifications of Olpinski index [10]

Sz_a (^{0}C/min)	**Risk Rating**
>180	Very Highly Susceptible
80-180	Highly susceptible
40-80	Moderately susceptible
0-40	Poorly susceptible
<80	Poor liability to S.H. risk
>120	High liability to S.H. risk
80-120	Moderate liability to S.H. risk
39-193 °C/min For Indian coals	

CHAPTER – 4

RESULTS

ABSTRACT OF EXPERIMENTAL TECHNIQUES

1. Proximate analysis

A. Determination of moisture

Amount of coal: 1 g coal

Size of coal:-72 mesh size

Heating time: 1.5 hours at 110°C

B. Determination of volatile matter

Amount of coal: 1 g of coal

Size of coal: -72 mesh size

Heating time: 7 minutes at 925°C

C. Determination of ash

Amount of coal: 1 g of coal sample

Size of coal: - 72 mesh size

Heating time: 30 minutes at 450°C and 60 minutes at 850° C

2. Crossing Point Temperature

Amount of coal: 4 g of coal sample

Size of coal: -100+200 mesh size

Heating rate: 1˚C/min

Oxygen flow rate: 80 ml/min.

3. Olpinski Index

Amount of coal: 1g of coal sample

Size of coal: -72 mesh size

Oxygen flow rate: 80 ml/min

The nine coal samples were collected from different coalfields of India, four from MCL and five from SECL and are listed in the following table.

Table 4.1 List of coal samples

Sr No.	Name of the Coal Sample	Name of the Coalfield
1	MCL-1	MAHANADI COALFIELDS LIMITED
2	MCL-2	
3	MCL-3	
4	MCL-4	
5	SECL-1	SOUTH EASTERN COALFIELDS LIMITED
6	SECL-2	
7	SECL-3	
8	SECL-4	
9	SECL-5	

The parameters of the proximate analysis are determined using Indian standard procedure of all collected coal samples after preparation as per the experimental requirements and the results of the proximate analysis after calculation are listed below:

Table 4.2 Results of proximate analysis

Sample	Moisture (%)	Volatile matter (%)	Ash (%)	Fixed carbon (%)
MCL-1	9.95	53.45	19.74	16.86
MCL-2	12.73	44.125	10.89	32.255
MCL-3	5.79	45.96	10.155	38.095
MCL-4	11.705	22.48	22.74	43.075
SECL-1	7.32	32.38	25.32	34.98
SECL-2	8.895	38.165	19.29	33.65
SECL-3	12.635	31.135	18.13	38.13
SECL-4	7.76	32.74	17.07	42.43
SECL-5	10.025	48.3	20.37	21.305

The crossing point temperature of all the coal samples are determined using the CPT apparatus designed by Prof Ramlu in an air medium bath and the Olpinski index also determined using the standard Olpinski index apparatus and the results are listed below:

Table 4.3 Results of CPT and Olpinski index

Sr No.	Sample	CPT(°C)	Sz_a (°C/min)	Sz_b (°C/min)
1	MCL-1	165	33	39.87
2	MCL-2	173	28	31.42
3	MCL-3	168	30	33.39
4	MCL-4	164	34	44
5	SECL-1	163	36	48.2
6	SECL-2	159	50	61.92
7	SECL-3	157	52	63.52
8	SECL-4	162	36	45.82
9	SECL-5	156	56	70.32

Based on the classification of CPT and Olpinski index (Table 3.1 and 3.2), the fire risk to spontaneous heating of all the coal samples are calculated and are listed in the following table.

Table 4.4 Fire risk rating

SL NO	SAMPLE	CPT RISK		OLPINSKI INDEX RISK	
		CPT(°C)	FIRE RISK	Sz_a(°C/min)	FIRE RISK
1	MCL-1	165	LOW	32	LOW
2	MCL-2	173	LOW	28	LOW
3	MCL-3	168	LOW	30	LOW
4	MCL-4	164	LOW	34	LOW
5	SECL-1	163	LOW	36	LOW
6	SECL-2	159	MEDIUM	50	MEDIUM
7	SECL-3	157	MEDIUM	52	MEDIUM
8	SECL-4	162	LOW	36	LOW
9	SECL-5	156	MEDIUM	56	MEDIUM

CHAPTER – 5

ANALYSIS

Correlation analysis was done between proximate parameters of coal (moisture, volatile matter, ash) and CPT and Olpinski index using the Microsoft Excel Package. In a different results-oriented user interface, Microsoft Office Excel 2 provides powerful tools and features that one can use to analyze, share, and manage data with ease. Commands and features that were often buried in complex menus and toolbars are now easier to find on task-oriented tabs that contain logical groups of instructions and facilities.

Table 5.1 Results of Correlation

Parameters of Proximate analysis	CPT	Sz_a
Moisture	-0.01403	0.187232
Volatile Matter	0.211145	-0.06257
Ash	0.052876	0.232837

FINDINGS

1. Moisture and CPT have positive correlation, which implies that increase in moisture content decreases the value of CPT.
2. Ash content has good positive correlation with CPT , Sz_a and Sz_b.
3. Volatile matter showing negative correlation with Sz_a and Sz_b, which indicates that increase in volatile matter decreases the Sz_a and Sz_b values.
4. CPT has a good correlation with volatile matter, so increase in volatile matter may increase the CPT.
5. Sz_a and volatile matter have good correlation

CHAPTER – 6

CONCLUSIONS

From the results obtained, correlation analysis and fire risk classification of CPT and Olpinski index the followings points are observed.

1. Highest moisture content is found in MCL-2 and SECL-3 also during field observation it was found that the seams are watery.

2. Results shows that SECL-3, SECL-2 and SECL-5 having CPT below 160°C and Sz_a less than 40°C/min, so these are moderately susceptible to spontaneous heating.

3. MCL-2, MCL-1, MCL-3, MCL-4, SECL-1 and SECL-4 has CPT value above 160°C and Sz_a below 40°C/min, which indicates that these are poorly susceptible to spontaneous heating

4. SECL-5 has highest Sz_a (Olpinski index) and lowest CPT, so it is more susceptible to spontaneous heating than other samples.

5. From the correlation graphs it was found that both CPT and Sz_a have highest R^2 =0.0542 with ash

6. MCL-2 has lowest Sz_a, so it is less susceptible to spontaneous heating.

7. Table 5.1 shows that with increase in moisture content, the CPT decreases and Sz_a increases, so moisture has a positive effect on spontaneous heating.

8. As Sz_a has good correlation with almost all the values, so Olpinski index can be used for better assessment of spontaneous heating

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APPENDIX-1

CPT CURVES

The graphical presentation of crossing point temperature of all the collected coal samples are shown in the following figures:

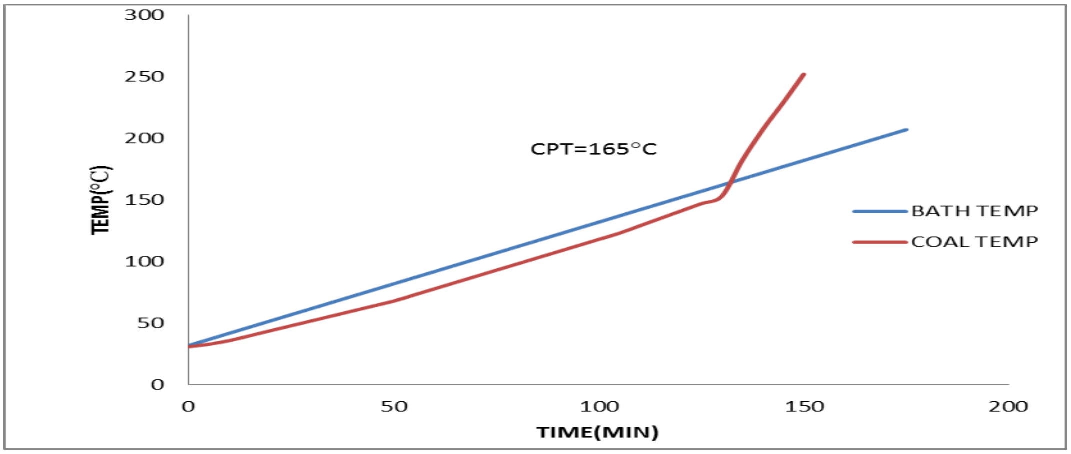

Fig. A-1 CPT curve of MCL-1

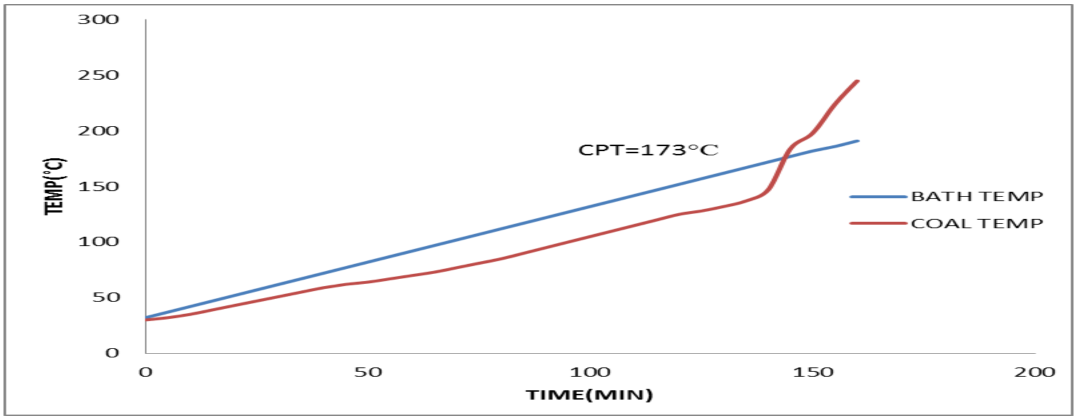

Fig. A-2 CPT curve of MCL-2

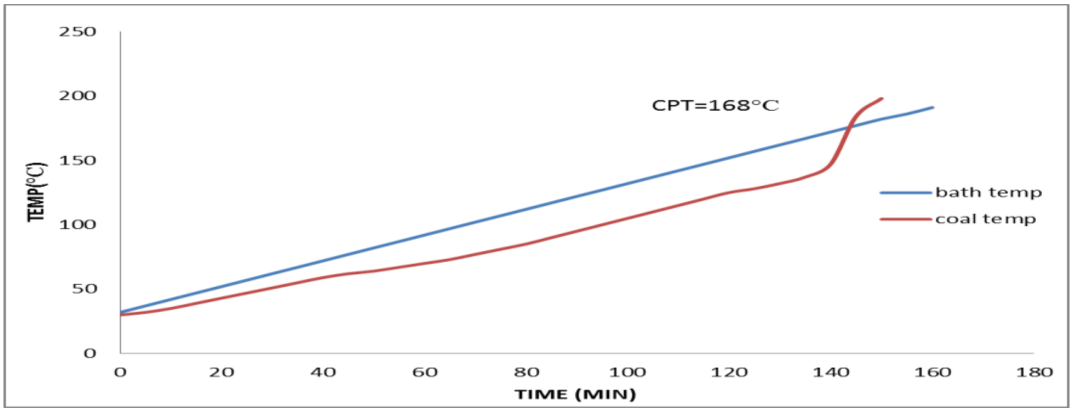

Fig. A-3 CPT curve of MCL-3

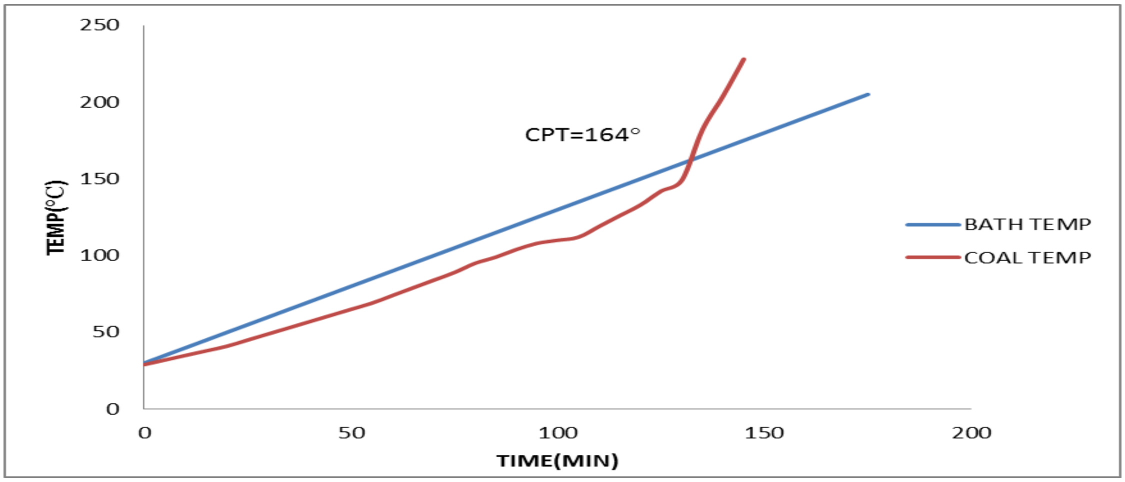

Fig. A-4 CPT curve of MCL-4

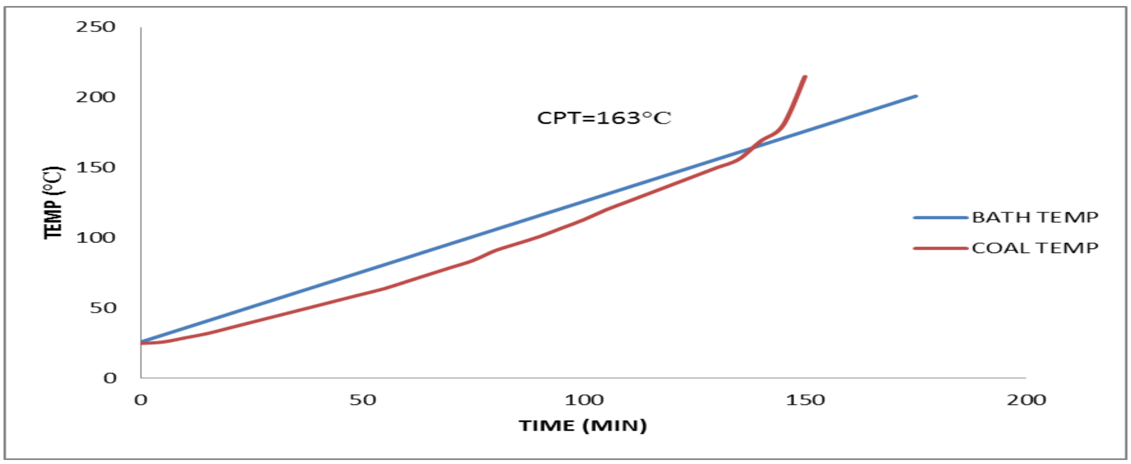

Fig. A-5 CPT curve for SECL-1

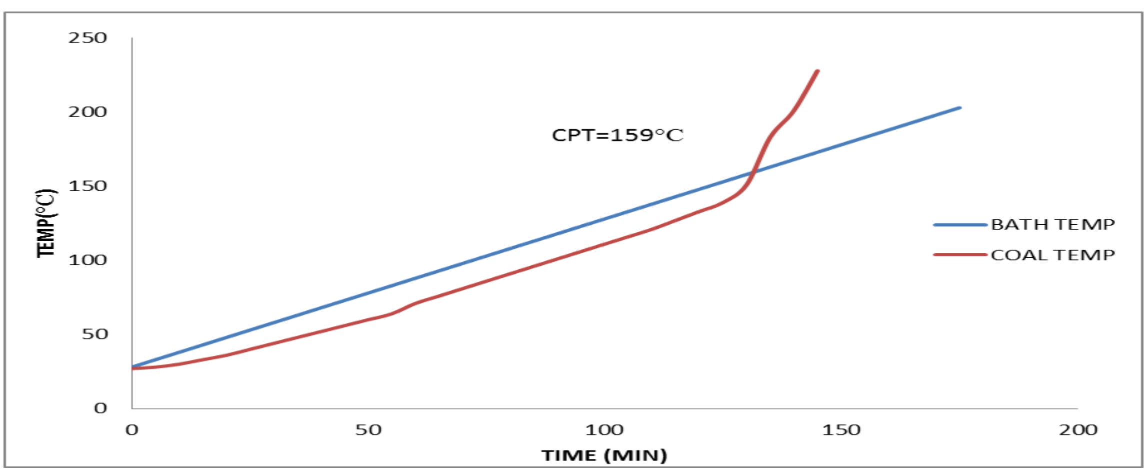

Fig. A-6 CPT curve for SECL-2

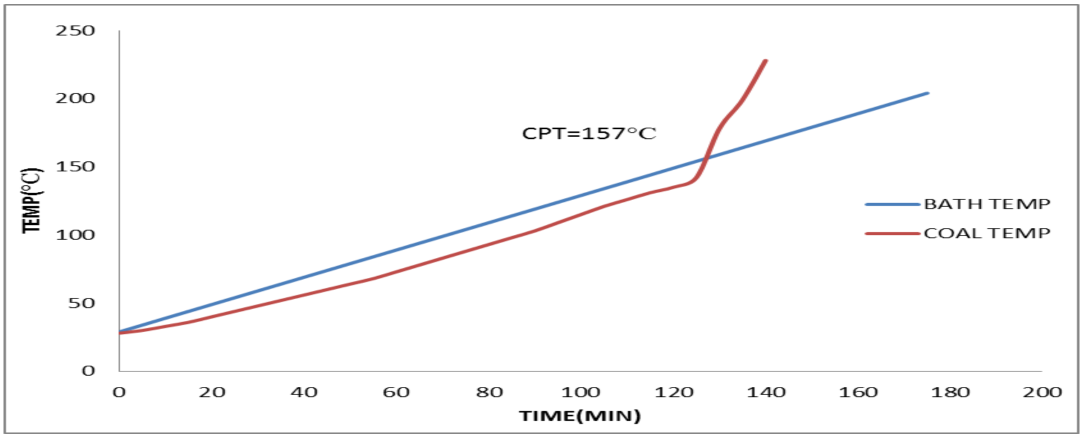

Fig. A-7 CPT curve for SECL-3

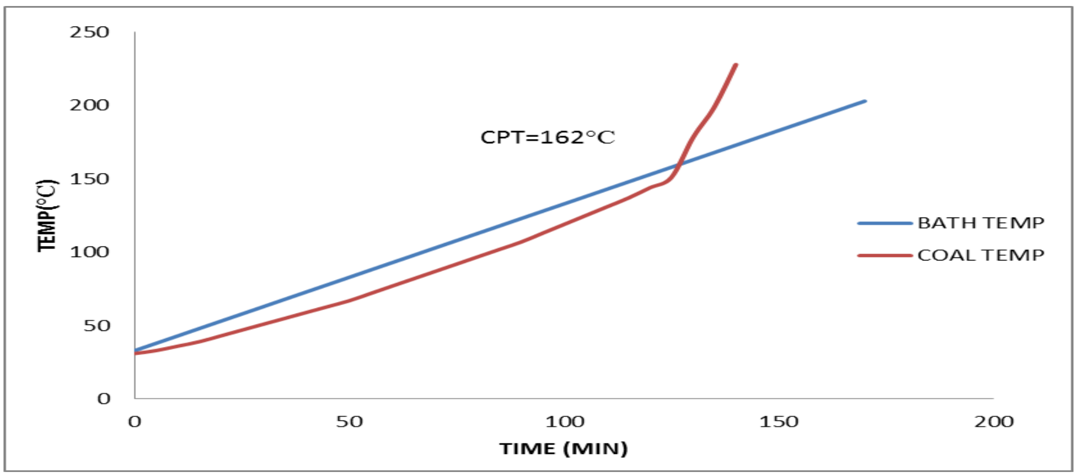

Fig. A-8 CPT curve for SECL-4

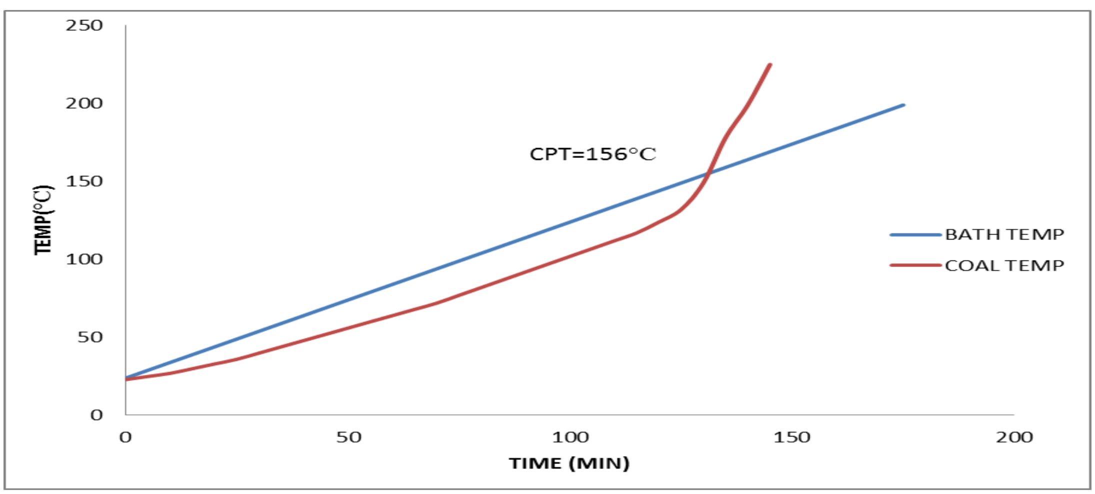

Fig. A-9 CPT curve for SECL-5

APPENDIX-2

OLPINSKI INDEX CURVES

The graphical presentation of Olpinski index of all the coal samples are shown in the following figures:

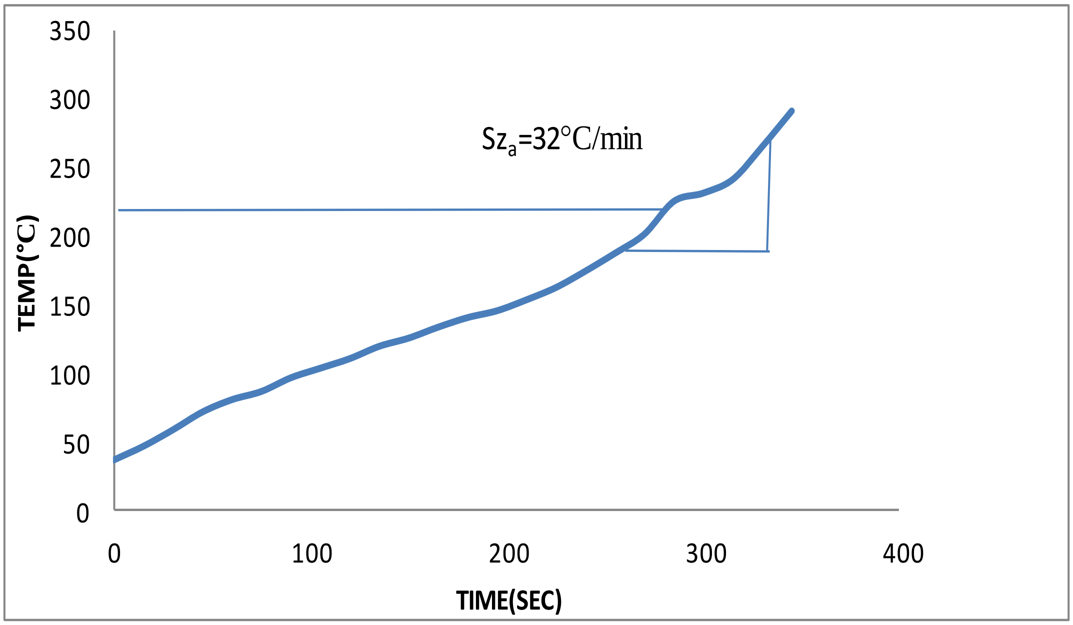

Fig. B-1 Olpinski curve of MCL-1

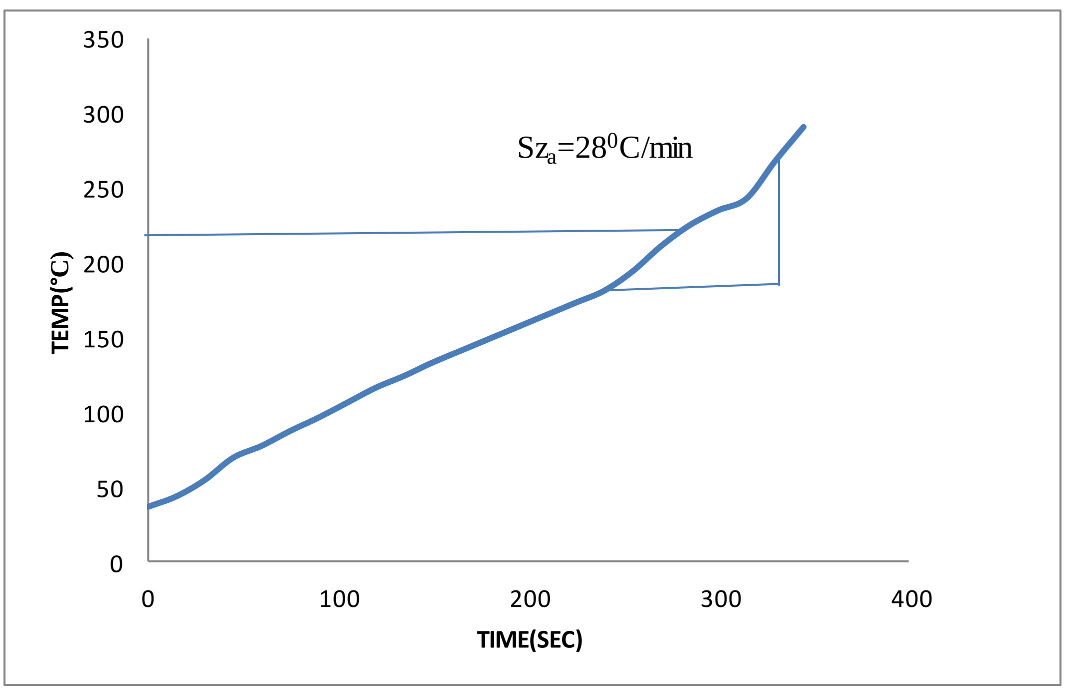

Fig. B-2 Olpinski curve of MCL-2

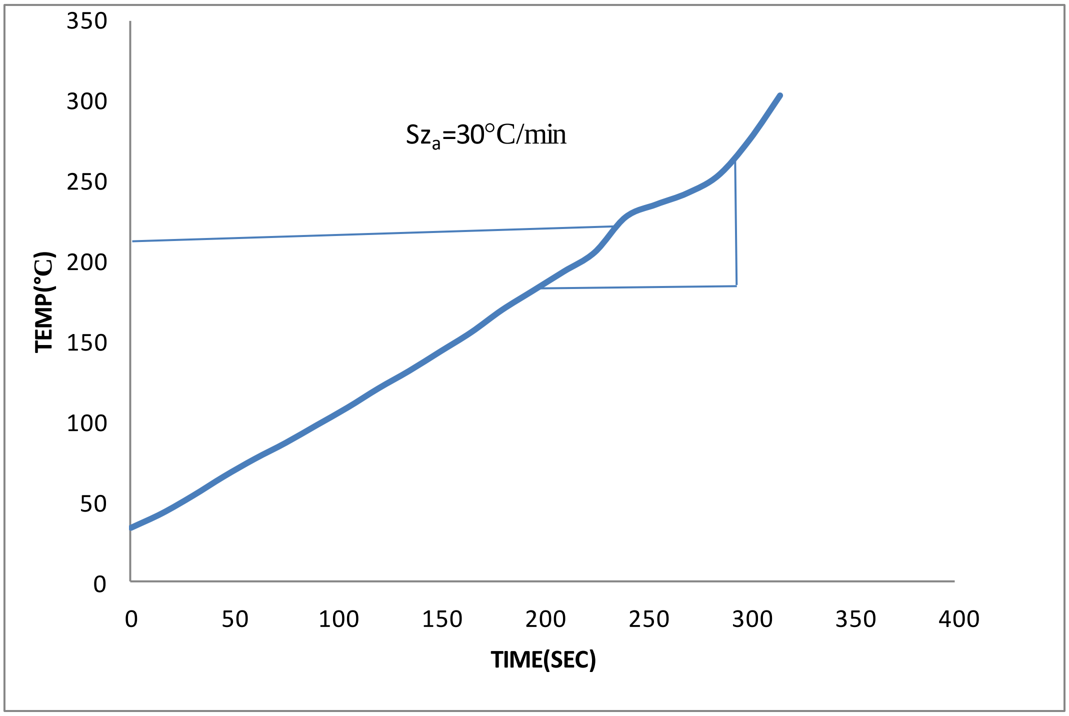

Fig. B-3 Olpinski curve of MCL-3

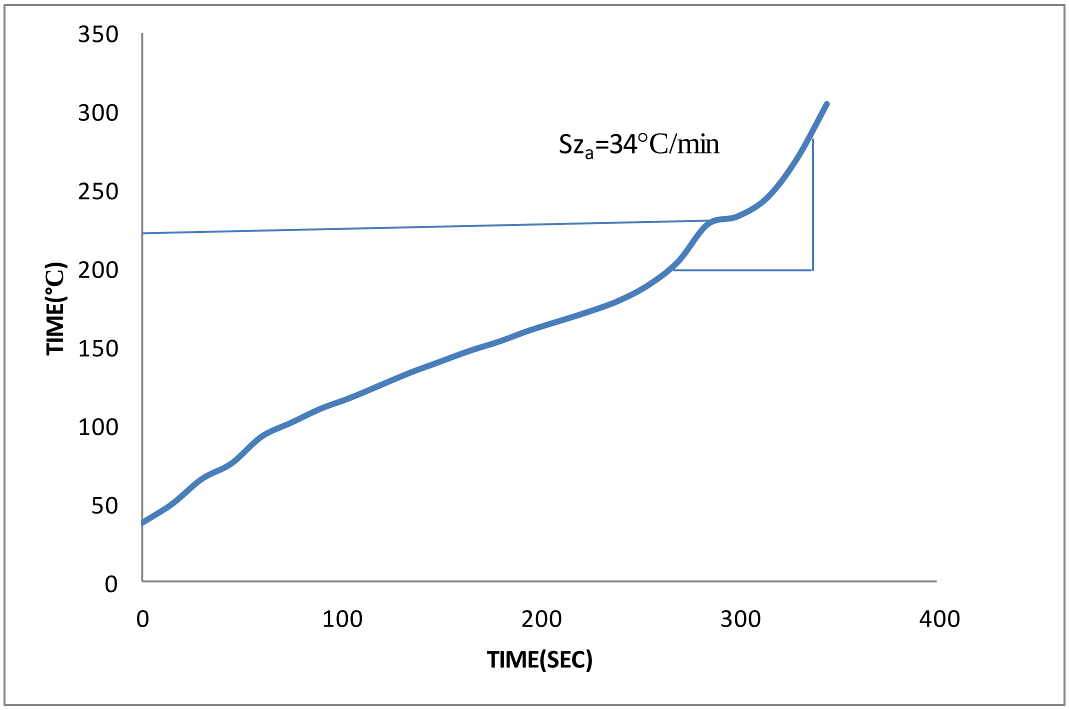

Fig. B-4 Olpinski curve of MCL-4

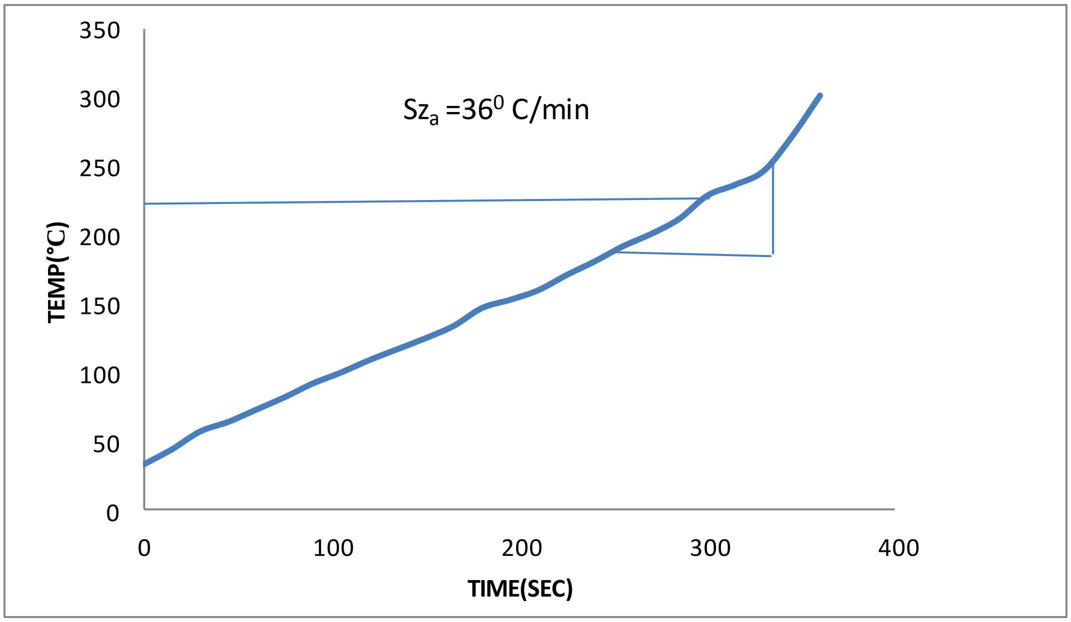

Fig. B-5 Olpinski curve of SECL-1

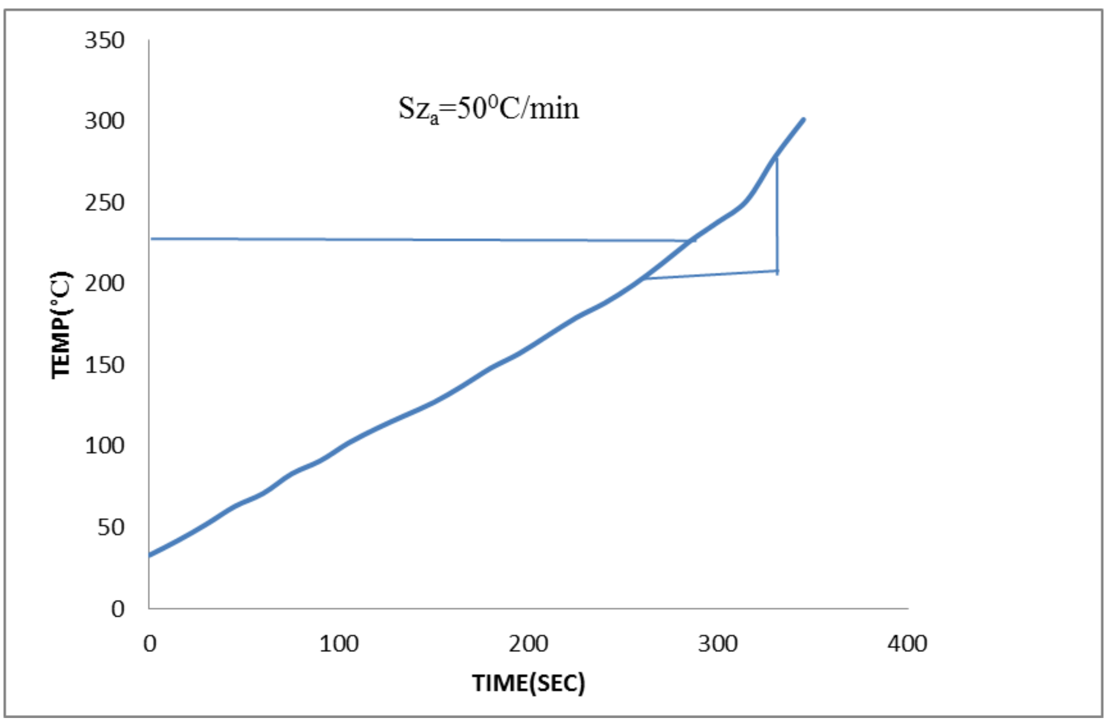

Fig. B-6 Olpinski curve of SECL-2

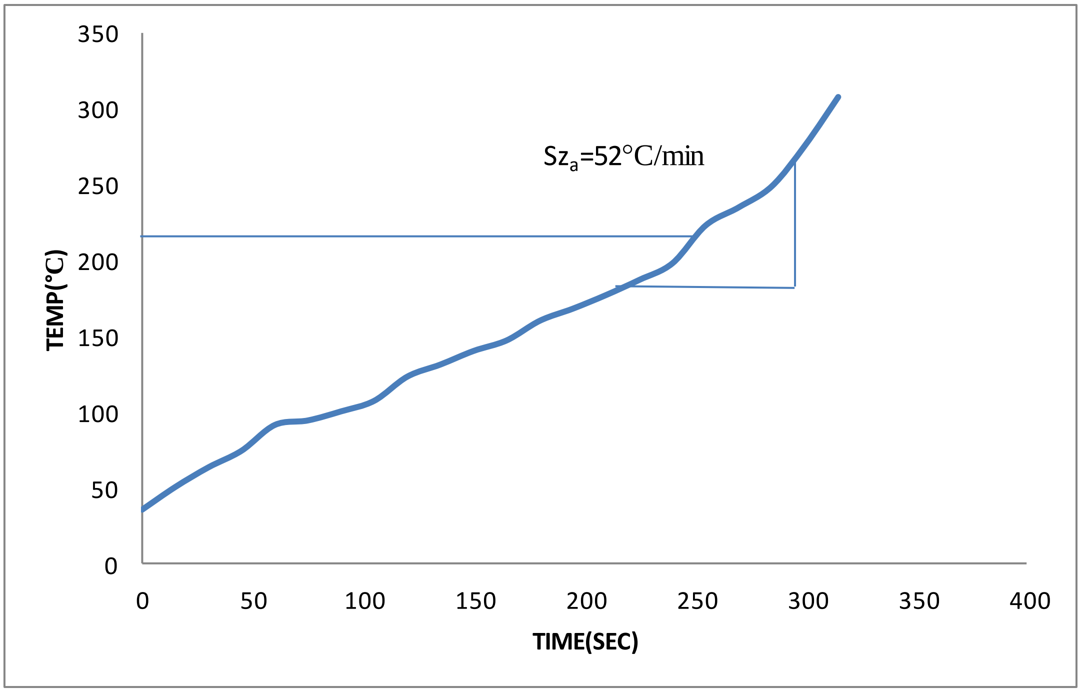

Fig. B-7 Olpinski curve of SECL-3

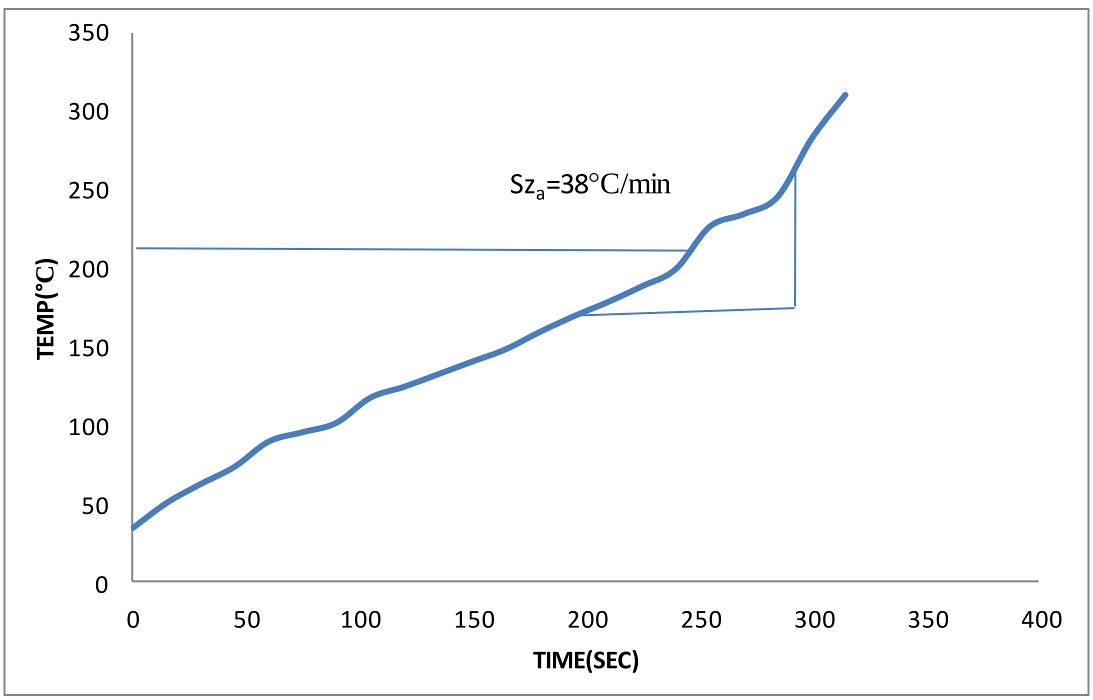

Fig. B-8 Olpinski curve of SECL-4

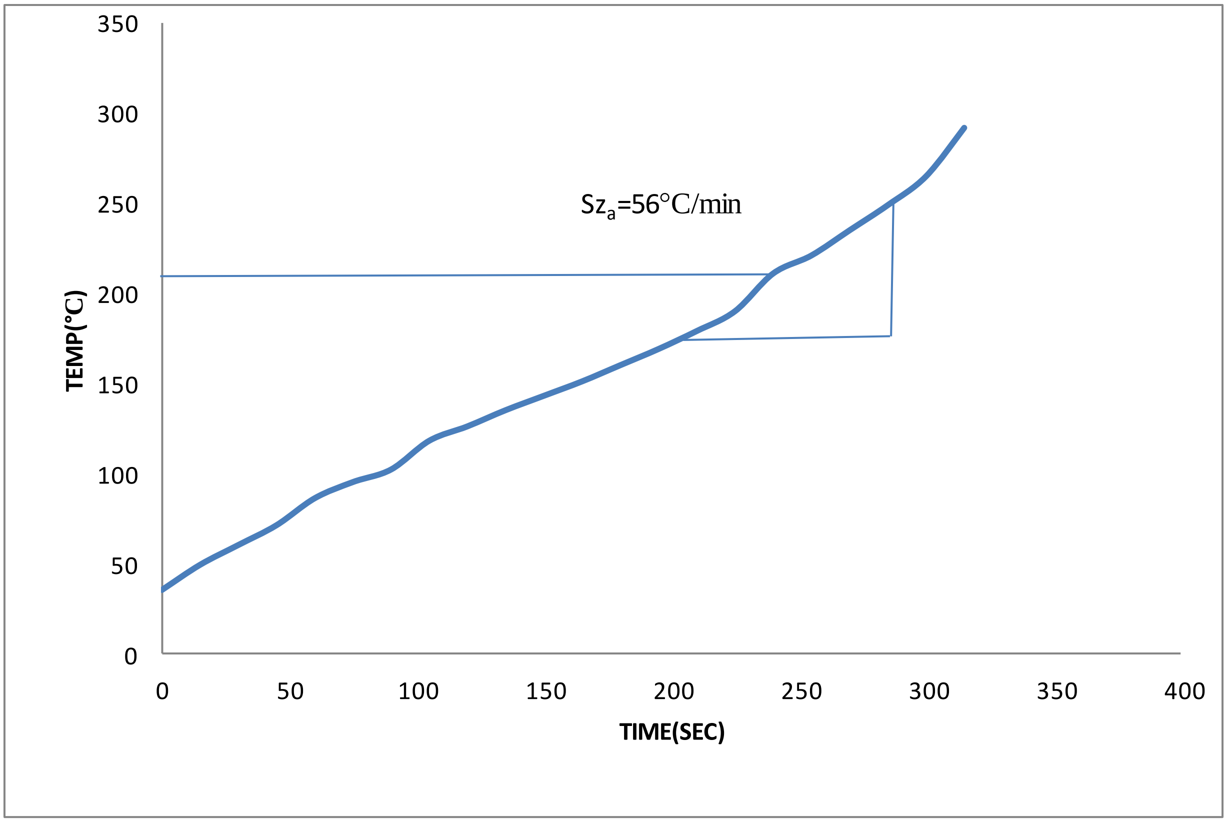

Fig. B-9 Olpinski curve of SECL-5

APENDIX-3

CORRELATION CURVES

The correlation analysis between the parameters of proximate analysis and CPT and Olpinski Index are shown in the following figures:

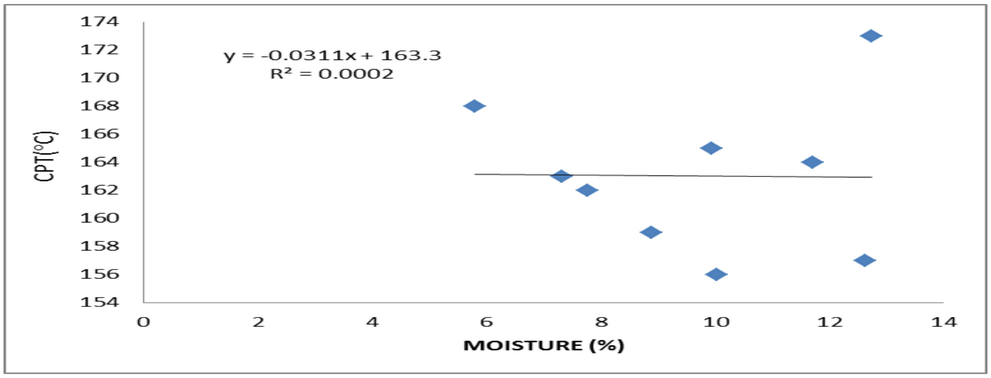

Fig. C-1 Correlation curve between Moisture and CPT

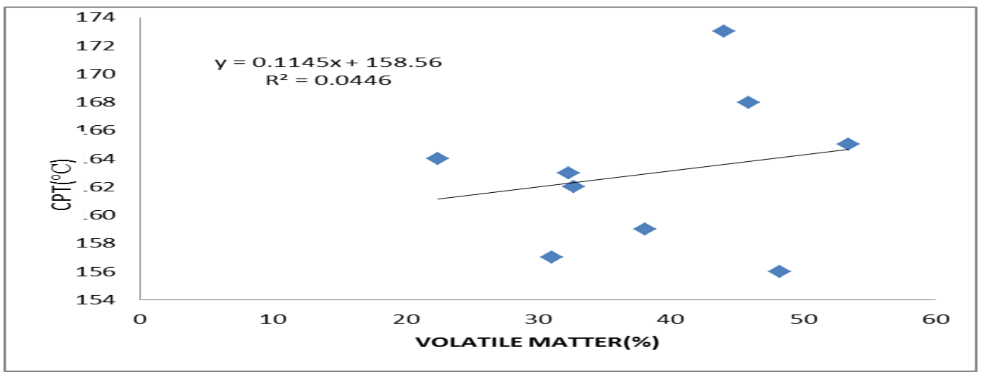

Fig. C-2 Correlation curve between Volatile matter and CPT

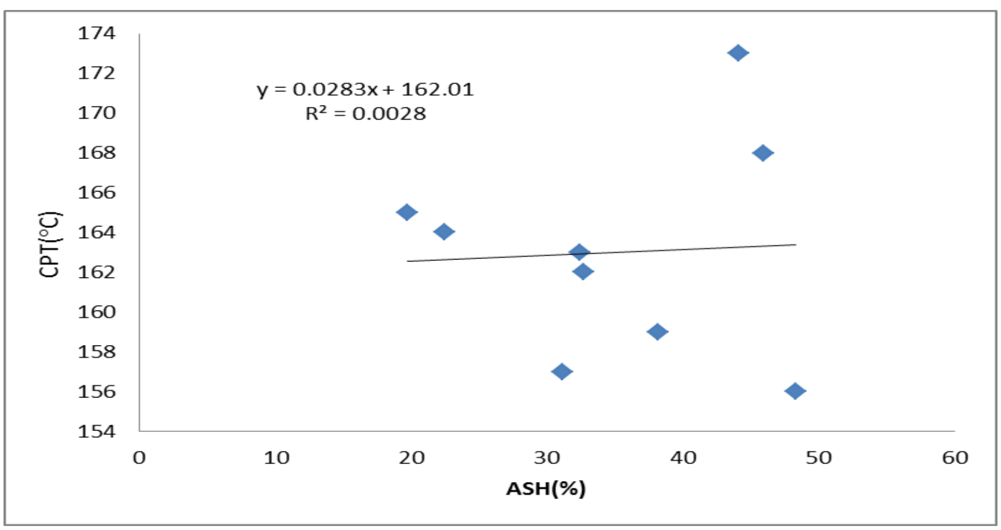

Fig. C-3 Correlation curve between Ash and CPT

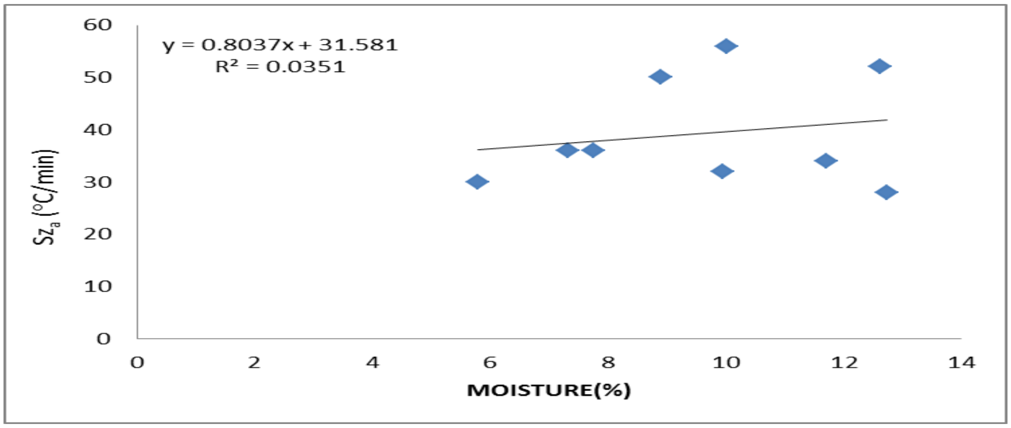

Fig. C-4 Correlation curve between Moisture and Sz$_a$

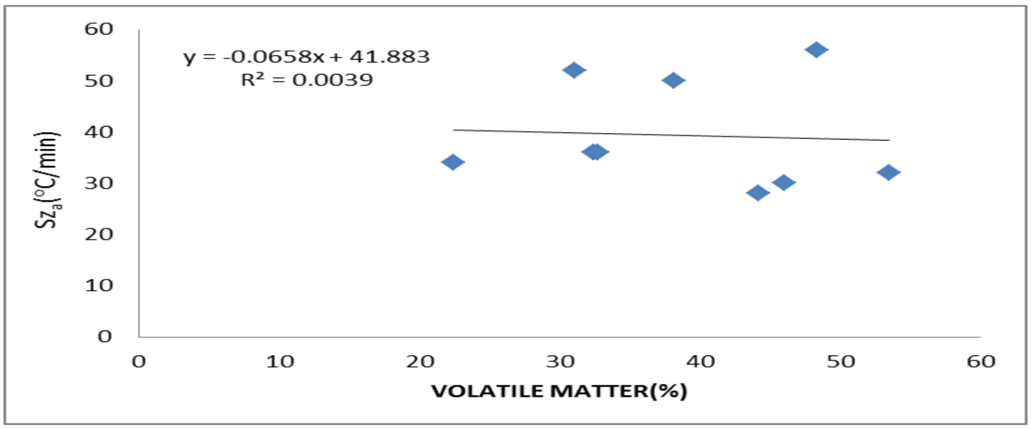

Fig. C-5 Correlation curve between Volatile matter and Sz$_a$

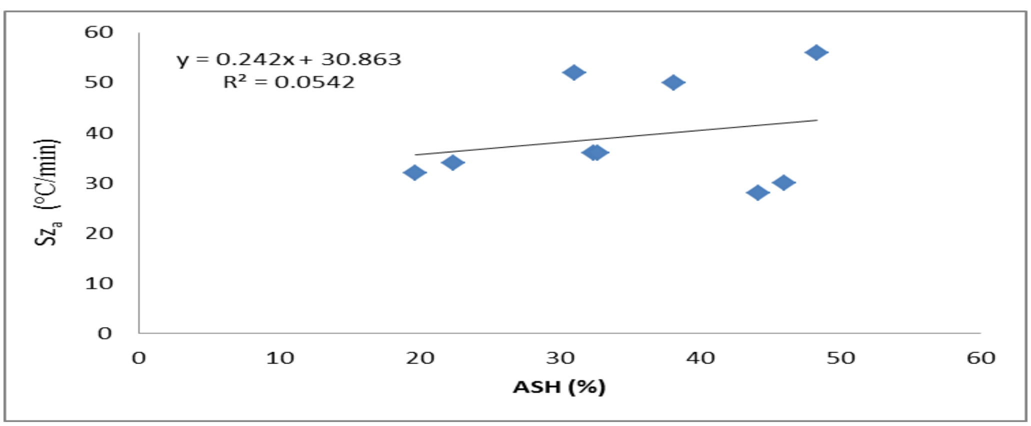

Fig. C-6 Correlation curve between Ash and Sz$_a$

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